

REMARKS

This is a full and timely response to the Office Action mailed June 29, 2007.

By this Amendment, claims 1-10 have been amended to put the claims in better form under U.S. practice, to address the rejection under 35 U.S.C. §112, second paragraph, and to more particularly define the present invention. Support for the claim amendments can be found variously throughout the specification and the original claims. Thus, claims 1-10 are pending in this application.

In view of this amendment and the following remarks, Applicant believes that all pending claims are in condition for allowance. Reexamination and reconsideration in light of the above claims and the following remarks is respectfully requested.

Rejection under 35 U.S.C. §112

Claims 1-10 are rejected under 35 U.S.C. §112, second paragraph, for alleged indefiniteness. Applicant respectfully traverses this rejection.

However, in the interest of expediting the allowance of the present application, Applicant has amended claims 1-10 to address the Examiner concerns in item 1 of the Office Action. More specifically, claims 1 and 4 have been amended to more particularly define the present invention, and claims 9 and 10 have been amended so that claims 1 and 4 provide proper antecedent basis for these claims.

Thus, in view of the amendments to the claims, withdrawal of this rejection is respectfully requested.

Rejection under 35 U.S.C. §102

Claims 1-10 are rejected under 35 U.S.C. §102(b) as allegedly being anticipated by JP 2000-243132 (hereinafter JP'132). Applicant respectfully traverses this rejection.

To constitute anticipation of the claimed invention under U.S. practice, the prior art reference must literally or inherently teach each and every limitation of the claims. Here, in this case, based on Applicant's review of the cited reference and the Examiner's comments, Applicant

submits that all of the limitations of the claims are not taught in JP'132, with particular emphasis on the limitations "*core materials disposed on the surface of the base particle*", "*wherein the core material is composed of a conductive material different from a conductive material composing said conductive film or conductive films*", and "*core materials disposed on the surface of the base particle and made of conductive material*".

The present invention is directed to a conductive particle having core materials as shown in Figure 7 (see core material 3 in the Figure). The core material 3 is disposed on the surface of the base particle 2 for forming a projection 5b after formation of the conductive film or films 4 and/or 5.

In contrast, the conductive particle of JP'132 does not have such a core material for forming a projection. As seen from the machine translation of JP'132 submitted herewith, paragraph [0008] clearly describes that the conductive particle has a spherical core material as a base particle and the Ni or Ni alloy coating is formed on the surface of the spherical core material. The projections are formed on the outermost surface of the coating by plating of Ni or Ni alloy. In particular, the projections are formed together with the coating and thus, **the projection are made of the same material as the coating**.

Accordingly, it is clear that JP'132 does not teach or suggest the "core material" of the present invention, which is made of a conductive material different from the conductive film or films. Thus, for these reasons, withdraw of this rejection is respectfully requested.

CONCLUSION

For the foregoing reasons, all the claims now pending in the present application are believed to be clearly patentable over the outstanding rejections. Accordingly, favorable reconsideration of the claims in light of the above remarks is courteously solicited. If the Examiner has any comments or suggestions that could place this application in even better form, the Examiner is requested to telephone the undersigned attorney at the below-listed number.

Dated: October 1, 2007

Respectfully submitted,

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PATENT ABSTRACTS OF JAPAN

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(21)Application number : 11-043005 (71)Applicant : NIPPON CHEM IND CO LTD
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(54) CONDUCTIVE ELECTROLESS PLATING POWDER, MANUFACTURE THEREOF, AND CONDUCTIVE MATERIAL MADE THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a favorable electric conductivity against connection between conductor patterns or electrodes having oxide coating on the surface by providing minute projections of specific length on the coating outermost surface layer, and substantially making the coating and the minute projections to be a continuous coating.

SOLUTION: By executing an electroless Ni plating method on the surface of particles, Ni or Ni alloy coating is formed. For Ni alloy, Ni-P, Ni-B alloy, or the like is mentioned. The coating outermost surface is provided with minute projections of 0.05-4 μm , and the size of the minute projection is set under 20% against average particle diameter. The minute projections are required to exist one or more in $(D/2)^2\text{m}^2$ (where D is the average diameter of electroless plating powder particles) on the surface on one piece of electroless plating particle. The minute projections and Ni-coating are formed into continuous coating, the minute projection is not detached by ultrasonic wave or the like, and hence it has excellent adhesion.

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CLAIMS

[Claim(s)]

[Claim 1] They are the conductive nonelectrolytic plating fine particles which have a 0.05-4-micrometer minute projection in this coat outermost layer, and are characterized by this coat and this minute projection being continuation coats substantially in the conductive nonelectrolytic plating fine particles which formed nickel or a nickel alloy coat by the nonelectrolytic plating method on the spherical core material particle front face whose mean particle diameter is 1-20 micrometers.

[Claim 2] Minute projections are conductive nonelectrolytic plating fine particles according to claim 1 which exist on the front face of one nonelectrolytic plating fine-particles particle in [one or more] 2 ($D/2$) micrometer² (however, D average diameter of a nonelectrolytic plating fine-particles particle).

[Claim 3] Conductive nonelectrolytic plating fine particles in which the gilding coat was formed on conductive nonelectrolytic plating fine particles according to claim 1.

[Claim 4] Claim 1 which a spherical core material particle becomes from a resin particle thru/or conductive nonelectrolytic plating fine particles of three given in any 1 term.

[Claim 5] Catalyst-sized down stream processing which this is returned [down stream processing] and makes a spherical core material particle front face support palladium after making a spherical core material particle front face catch palladium ion, manufacture approach [of the conductive nonelectrolytic plating fine particles which consist of performing both the processes of following A process and B process at least after that]: -- A process: -- the aquosity slurry of a spherical core material -- nickel salt -- A nonelectrolytic-plating process, B process which are added to the nonelectrolytic-plating bath containing a reducing agent, a complexing agent, etc.: The nonelectrolytic plating process which divides the constituent of nonelectrolytic plating liquid into the aquosity slurry of a spherical core material at at least 2 liquid, and adds each that it is simultaneous and with time.

[Claim 6] The manufacture approach of the conductive nonelectrolytic plating fine particles according to claim 5 which subsequently carry out B process after carrying out the introduction A process.

[Claim 7] The manufacture approach of the conductive nonelectrolytic plating fine particles which add performing C process which performs gilding processing further to the manufacture approach of conductive nonelectrolytic plating fine particles according to claim 5 or 6.

[Claim 8] The conductive ingredient which consists of claim 1 thru/or conductive nonelectrolytic plating fine particles of four given in any 1 term.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a conductive ingredient at the conductive nonelectrolytic plating fine particles used for the electroconductive glue for carrying out flow adhesion of the connection circuit which counters in detail the conductive nonelectrolytic plating fine particles used for microelectrode junction of for example, electronic equipment, and its manufacture approach list about the conductive ingredient which consists of these plating fine particles, the anisotropy electric conduction film, anisotropy electric conduction adhesives, etc., and its manufacture approach list.

[0002]

[Description of the Prior Art] Conventionally, as conductive fine particles used for electroconductive glue, the anisotropy electric conduction film, anisotropy electric conduction adhesives, etc., the conductive plating fine particles which covered metals, such as nickel and nickel-gold, copper, gold, silver, and solder, with nonelectrolytic plating, vacuum deposition, etc. on the front face of carbon system; resin core material particles, such as metal powder, carbon powder, such as nickel, copper, silver, gold, and solder, and carbon fiber, a carbon flake, are known. The conductive fine particles using the above-mentioned metal powder have large specific gravity, and the application for which a configuration is an indeterminate form, and sedimentation or decentralization is very difficult for, and is used in case it is mixed and used for various matrix materials, since particle size distribution are large is limited.

[0003] The conductive fine particles using the above-mentioned carbon system powder have the low conductivity of carbon itself, and it is not used for the application of which conductive high ability and high-reliability are required. The conductive fine particles using the above-mentioned conductive plating fine particles Generally core material powder is immersed in the plating liquid prepared beforehand, and it is manufactured by the approach of suspending the post-reaction which was defined by the experiential guess and which carried out the time amount plating reaction. Although that to which the nonelectrolytic plating powder obtained by this approach has a projection in a front face is easy to be obtained, since the autolysis of plating liquid arises in the case of powder and a granule with a big specific surface area of a galvanized core material, the nonelectrolytic plating powder obtained becomes what the detailed nickel decomposition product mixed. Moreover, in order to form firm floc, with physical means etc., if it cracks, floc will break, and the phenomenon which a non-covering surface exposes is caused.

[0004] As a nonelectrolytic plating means against the powder-like core material which solved such a problem, the conductive filler which the detailed metal particles by the nonelectrolytic plating method become from the nonelectrolytic plating powder by which sediment formation was carried out as a dense and substantial continuation coat is shown in the base material front face of the quality of organic or minerals which for example, these people developed previously (JP,1-242782,A). Sediment formation is carried out as a continuation coat that the detailed metal particles of the nonelectrolytic plating powder obtained by the above-mentioned approach by which plating formation was carried out are dense to core material powder, and substantial, and the coat configuration did not become phyma-like, but when it excels in smooth nature and was used for electroconductive glue, the anisotropy electric conduction film, anisotropy electric conduction adhesives, etc., it became possible to give the outstanding high conductivity ability.

[0005] However, since the front face is smooth (for example, since the 3-9nm oxide film usually exists in the aluminum circuit pattern front face in case it is used for electroconductive glue on which the wiring substrate with which the aluminum circuit pattern was formed is pasted up after the aluminum circuit pattern has met), the nonelectrolytic plating powder obtained by the above-mentioned approach cannot tear the oxide film, and since a touch area is also small, it may be unable to acquire good conductivity.

[0006] Moreover, the method of manufacturing a conductive particle is indicated by JP,4-36902,A by performing metal plating on the front face of a non-conductive particle which had a projection in the front face. or [however, / making the child particle of the same quality of the material as the particle (mother particle) front face which the above-mentioned conductive particle has the description in a core material, and shows the shape of smooth surface type or the different quality of the material adhere using adhesives] -- or welding is carried out directly -- Or since it is obtained by making a projection form and performing metal plating on a particle front face by the approach of evaporating a solvent etc., putting into the container turning around a mother particle, making a child particle adhere to a particle front face, and rotating a container, By a plating head end process etc., it has the fault of a child particle ****ing easily by sonication used for distribution, dispersion arises in the surface state after plating, and always good conductivity cannot be acquired.

[0007]

[Problem(s) to be Solved by the Invention] This invention solves the above-mentioned problem and the purpose is in

offering the conductive ingredient which serves as industrially conductive nonelectrolytic plating fine particles which have good conductivity from these nonelectrolytic plating fine particles at the advantageous manufacture approach list to inter-electrode connection between the conductor patterns which have an oxide film on a front face.

[0008]

[Means for Solving the Problem] That is, this invention offers the conductive nonelectrolytic plating fine particles which have a 0.05-4-micrometer minute projection in this coat outermost layer, and are characterized by this coat and this minute projection being continuation coats substantially in the conductive nonelectrolytic plating fine particles which formed nickel or a nickel alloy coat by the nonelectrolytic plating method on the spherical core material particle front face whose mean particle diameter is 1-20 micrometers. This invention offers the manufacture approach of the conductive nonelectrolytic plating fine particles which serve as catalyst-ized down stream processing which this is returned [down stream processing] and makes a spherical core material particle front face support palladium from performing both the processes of following A process and B process at least after that further again, after making a spherical core material particle front face catch palladium ion.

A process: The nonelectrolytic plating process which divides the constituent of nonelectrolytic plating liquid into the aquosity slurry of a nonelectrolytic plating process and B process:spherical core material which adds the aquosity slurry of a spherical core material to the nonelectrolytic plating bath containing nickel salt, a reducing agent, a complexing agent, etc. at at least 2 liquid, and adds each that it is simultaneous and with time.

This invention offers the conductive ingredient which used the aforementioned conductive nonelectrolytic plating fine particles further again.

[0009]

[Embodiment of the Invention] The conductive nonelectrolytic plating fine particles which this invention tends to offer In the nonelectrolytic plating fine particles by which mean particle diameter formed preferably 1-20 micrometers of nickel or nickel alloy (it may only explain as nickel hereafter) coats by the nonelectrolytic plating method on the 3-10-micrometer spherical core material particle front face It has a 0.05-4-micrometer minute projection in the nickel coat outermost layer, and a nickel coat and a minute projection are characterized by being a continuation coat substantially on a configuration. As for these plating fine particles, nickel or a nickel alloy coat is formed in the particle front face by the non-electrolyzed nickel-plating method. There are nickel-Lynn, a nickel-boron alloy, etc. as a nickel alloy.

[0010] This front face has a 0.05-4-micrometer minute projection, and it is suitable for the magnitude of this minute projection that it is 20% or less to the mean particle diameter of nonelectrolytic plating fine particles. For example, in the case of 5-micrometer mean particle diameter, a minute projection is 1 micrometer or less, and, in the case of 10 micrometers, it is set to 2 micrometers or less. A minute projection whose reason for making a minute projection into 20% or less of mean particle diameter exceeds 20% is because manufacture is difficult substantially. The magnitude of the minute projection has the plating thickness and relation which are mentioned later, and, as for the magnitude, only the thing of about 10-time magnitude is obtained at the maximum to plating thickness. For example, when plating thickness is 0.2 micrometers, as for the magnitude of a minute projection, a thing 2 micrometers or less is formed. This thickness is checked by the chemical analysis, and the magnitude of a minute projection can be checked with an electron microscope photograph.

[0011] Although especially the quality of the material of this minute projection is not limited, it is desirable that they are nickel or a nickel alloy. Although this minute projection needs to recognize a large number existence on the front face of a nonelectrolytic plating fine-particles particle piece, it needs to exist more than the piece in at least $(D/2)^2$ 2-micrometer² (however, D average diameter of a nonelectrolytic plating fine-particles particle). The abundance of this minute projection can also be checked with an electron microscope photograph. Especially the configuration of this minute projection may not be limited and may be which configurations, such as the shape of the shape of a semicircle, and a cone, and a pyramid.

[0012] Although the conductive nonelectrolytic plating fine particles of this invention have the above projections, the structure forms a minute projection and nickel coat of nickel in a spherical core material particle by the non-electrolyzed nickel-plating method at coincidence. Although the structure consists of this minute projection and a nickel coat For example, after making the nucleus and nickel coat of a minute projection form on a spherical core material particle at coincidence, After the nickel coat was formed on object (b) which homogeneity and a continuation nickel coat form in this front face further, and a spherical core material particle, They are the object (Ha) with which the nickel coat is formed in object (**) by which the nucleus and nickel coat of a minute projection are formed in this front face at coincidence, and (**), object (d) by which the gilding coat is further formed in (**) - (Ha) a front face.

[0013] Since, as for each above-mentioned conductive nonelectrolytic plating fine particles, this minute projection is also growing with the nickel growth of anodic oxide film, the minute projection and the nickel coat are a continuation coat, and it becomes the description on structure for a minute projection not to **** with a supersonic wave etc. and to excel in adhesion. The nickel coat and minute projection which form this continuation coat can be checked by the cutting plane of this particle.

[0014] Although there will be especially no limitation in the quality of the material if spherical core material particles are fine particles insoluble in water, the shape of a ball is presented in appearance as description, and it is chosen from the fine particles of the minerals in which nonelectrolytic plating is possible, or the quality of organic. As inorganic spherical core material fine particles, the oxide (an inclusion is also included) of metal powder, a metal, or a nonmetal, the metal silicate containing aluminosilicate, metallic carbide, a metal nitride, a metal carbonate, a metal sulfate, metallic phosphorus acid chloride, metallic sulfide, metal acid chloride, a metal halogenide or carbon, glass powder, etc. can be illustrated.

[0015] As spherical core material fine particles of the quality of organic, for example Polyethylene (PE), a polyvinyl

chloride (PVC), A polyvinylidene chloride, polytetrafluoroethylene (PTFE), polypropylene (PP), Polystyrene (PS), a polyisobutylene (PIB), polyvinyl pyridine, Polyolefines, such as polybutadiene (BR), polyisoprene, and polychloroprene, A styrene-acrylonitrile copolymer (SAN), an AKURIRONIDARIRU-butadiene-styrene terpolymer (ABS), An ethylene-methacrylic-acid copolymer (ionomer), a styrene butadiene rubber (SBR), Nitrile rubber (NBR), an ethylene propylene elastomer, isobutylene isoprene rubber, Olefin copolymers, such as a thermoplastic olefin elastomer, polyacrylate, Acrylic-acid derivatives, such as polymethylmethacrylate (PMMA) and polyacrylamide, Polyvinyl acetate (PVA), polyvinyl alcohol (PVAL), a polyvinyl butyral (PVB), A polyvinyl formal (PVF), polyvinyl ether, a polyvinyl pyrrolidone, Polyvinyl compounds, such as a polyvinyl carbazole, flexible polyurethane foam, Polyurethane, such as rigid polyurethane foam and a polyurethane elastomer, Polyacetal, a polyethylene glycol (PEG), a polypropylene glycol (PPG), Ether polymers, such as an epoxy resin and polyphenylene oxide (PPO), Polyethylene terephthalate (PET), polybutylene terephthalate (PBT), Poly dihydroxy methylcyclohexyl terephthalate, cellulose ester, Polyester, such as unsaturated polyester, aromatic polyester, and a polycarbonate (PC), Polyamides, such as an aliphatic series polyamide, phenol resin, phenol-formaldehyde resin (PF), Formaldehyde resin (UF), melamine-formaldehyde resin (MF), Polyphenylene sulfide (PPS), polybenzimidazole (PBI), Benzoguanamine, a urea, thiourea, a melamine, acetoguanamine, The amino resin which consists of amino compounds, such as a cyanogen amide and an aniline, and aldehydes like formaldehyde, a paraformaldehyde, an acetaldehyde, and glyoxal, a fluorine-containing resin, nitril system resin, etc. can be mentioned. However, in these, the resin fine particles of the quality of organic are used suitably.

[0016] This core material particle is substantially spherical. Although a spherical particle means the thing [that the configuration near a globular form like an ellipse besides a perfect globular form may be included] substantially, it is so desirable that it is close to a globular form. As the shape of corpuscular character of a spherical core material particle, 1-20 micrometers of mean particle diameter are in the range of 3-10 micrometers preferably, and selection use of that whose CV value is 10% or less more preferably is carried out. In addition, a CV value means the coefficient of variation expressed with CV value %=(standard deviation)/(average) x100.

[0017] The nonelectrolytic plating layer formed in a spherical core material particle front face equipped with the shape of above corpuscular character may be the plating coat of nickel or a nickel alloy, and may be two or more sorts of double layer coats. In the case of a double layer coat, a nickel-***** coat is suitable. Although there are nickel-Lynn, nickel-boron, etc. and there are not Lynn in a coat and especially content of boron as a nickel alloy what is restricted, it is desirable that they are 5 or less % of the weight and 3 % of the weight or less, respectively. The reason limited to nickel or a nickel alloy coat is that there is profitableness which functions effectively as an interlayer who secures firm affinity with the upper plating coat layer in carrying out the double stratification of the gold to the top face, in being able to stick firmly with a spherical core material particle and being able to form the good nonelectrolytic plating layer of peeling resistance. Moreover, if it is made a nickel-***** coat, compared with a monolayer coat, conductive ability can be raised further.

[0018] The range of the non-electrolyzed nickel-plating thickness to form is 0.05-0.5 micrometers. In less than 0.05 micrometers, the homogeneity of a coat layer is missing and, moreover, conductive ability is inferior. If 0.5 micrometers is exceeded, particles will condense at a plating process, a bridge phenomenon will be produced, and dispersibility will be spoiled. Nickel thickness is average thickness which means thickness including a nickel coat and a minute projection, and is computed by the chemical analysis here.

[0019] The manufacture approach of the conductive nonelectrolytic plating fine particles concerning this invention has the description to combine the nonelectrolytic plating method of catalyst-ized down stream processing which this is returned [down stream processing] and makes a core surface support palladium, following A process after performing catalyst-ized processing, and B process, after making the front face of a spherical core material particle catch palladium ion.

[0020] A process is a nonelectrolytic plating process which adds the aquosity slurry of a spherical core material to the nonelectrolytic plating bath containing nickel salt, a reducing agent, a complexing agent, etc. At this A process, it is the approach by which the nucleus of a minute projection generates and formation of a nickel coat is made by it and coincidence by the autolysis of a plating bath happening to formation and coincidence of the nickel coat to a spherical core material particle top, and catching this autolysate on a core material particle front face at the time of formation of a nickel coat, since this autolysis is produced near the spherical core material particle. B process is a nonelectrolytic plating process which divides the constituent of nonelectrolytic plating liquid into the aquosity slurry of a spherical core material at at least 2 liquid, and adds each that it is simultaneous and with time (for example, continuously). At this B process, when a minute projection nucleus is on a spherical core material particle, growth and the nickel growth of anodic oxide film of a minute projection are performed to coincidence, and when there is no minute projection, formation of a continuation nickel coat is uniformly performed on a spherical core material particle.

[0021] Although the combination of the above-mentioned A process and B process has a method of performing A process subsequently and performing B process further etc. after performing B process at the beginning of the approach of subsequently performing A process after performing B process at the beginning of the approach of subsequently performing B process after performing A process at the beginning of **, and **, and **, especially this combination is not restricted.

[0022] In the approach of this invention, after making coincidence generate the nucleation of a minute projection, and formation of a nickel coat first on a spherical core material particle, the combination of ** in which homogeneity and a continuation nickel coat are made to form subsequently to this front face is desirable. Furthermore, in order to form a nickel-***** coat in this invention, it can manufacture by performing the nonelectrolytic plating C process of performing gilding processing on the spherical core material which formed the nickel coat in the combination of the above-mentioned

A process and B process.

[0023] If the concrete means of a nonelectrolytic plating method, for example, the combination of **, is explained, since nonelectrolytic plating will be performed by the drainage system, when there are almost no spherical core material fine particles at a hydrophilic property, it is necessary to carry out hydrophilization with an acid, alkali, etc. Selection of an acid or alkali is suitably chosen by the property of spherical core material fine particles. Subsequently, reforming processing which gives catalyst prehension ability to the front face of a spherical core material particle is performed. Catalyst prehension ability is a function which can catch palladium ion as a chelate or a salt on a spherical core material particle front face in catalyst-ized down stream processing, and a prehension function is one of those which generally have one sort of the amino group, an imino group, an amide group, an imide radical, a cyano group, a hydroxyl group, a nitrile group, or a carboxyl group, or two sorts or more on a spherical core material front face. Therefore, as spherical core material matter which has catalyst prehension ability, organic objects, such as epoxy system resin stiffened with an amino resin, nitril system resin, or an amino curing agent, can be mentioned, and these spherical core material fine particles are used suitable for the purpose of this invention.

[0024] Although surface treatment needs to give prehension ability when there is no catalyst prehension ability in the spherical core material itself, this reforming-ization can be performed using the epoxy system resin hardened with an approach, i.e., an amino-group permutation organosilane system coupling agent, and an amine system curing agent given in JP,61-64882,A. Catalyst-ized down stream processing fully distributes spherical core material fine particles in the thin aqueous acids of a palladium chloride, and makes palladium ion catch on a front face. The concentration of a palladium-chloride water solution is enough in the range of 0.05 – 1 g/L. Subsequently, after performing repulping washing, reduction processing of the palladium ion which the spherical core material particle front face was made to catch is carried out, and the front face of a spherical core material particle is made to catch palladium. Beforehand, this reduction processing makes spherical core material fine particles the shape of a slurry, and is performed by the approach of adding a reducing-agent water solution at the fully distributed place. As a reducing agent used, specific hypophosphite, a sodium borohydride, a boron hydride potassium, dimethylamine borane, a hydrazine, formalin, etc. are used. Although the addition of a reducing agent changes with specific surface area of a spherical core material, the range of 0.01 – 10 g/L is suitable for it to a slurry in general.

[0025] A nonelectrolytic plating A process makes water fully distribute preferably the spherical core material particle which performed catalyst-ized processing in the range of 5 – 300 g/L one to 500 g/L, and prepares an aquosity slurry. It can usually carry out to distributed actuation using stirring, high-speed stirring, a colloid mill, or shear distribution equipment like a homogenizer. Moreover, a supersonic wave may be used together to the above-mentioned distributed actuation. In addition, for distributed actuation, dispersants, such as a surfactant, may be added if needed. Subsequently, the spherical core material slurry which carried out distributed actuation to the nonelectrolytic plating bath containing nickel salt, a reducing agent, a complexing agent, various additives, etc. is added, and a nonelectrolytic plating A process is performed. At this nonelectrolytic plating A process, the nickel particle which serves as a nucleus of a minute projection at formation and coincidence of a nickel coat is formed on a spherical core material particle.

[0026] As nickel salt, a nickel chloride, a nickel sulfate, nickel acetate, etc. are used, and let the concentration be the range of 0.1 – 50 g/L. As a reducing agent, specific hypophosphite, dimethylamine borane, a sodium borohydride, a boron hydride potassium, a hydrazine, etc. are used, and the concentration is the range of 0.1-50g / L. As a complexing agent, the compound which has a complexing operation to nickel ion, such as amine acids, such as amino acid, such as carboxylic acids (salt), such as a citric acid, hydroxyacetic acid, a tartaric acid, a malic acid, a lactic acid, a gluconic acid or its alkali-metal salt, and ammonium salt, and a glycine, ethylenediamine, and alkylamine, other ammonium, EDTA, and a pyrophosphoric acid (salt), may be used, and these may be one sort or two sorts or more, for example. The concentration is the range of 5 – 50 g/L preferably one to 100 g/L. The range of pH of the desirable nonelectrolytic plating bath in this phase is 4-14. Although a reaction will start promptly and a nonelectrolytic plating reaction will be accompanied by generating of hydrogen gas if a spherical core material slurry is added, termination of a nonelectrolytic plating A process is considered as termination with the time of generating of the hydrogen gas no longer being accepted completely.

[0027] Subsequently, in B process, nonelectrolytic plating is performed by dividing into at least 2 liquid the requirements of each water solution of the nickel salt which constitutes nonelectrolytic plating liquid, specific hypophosphite, and a sodium hydroxide after the above-mentioned A process, and carrying out judgment addition of each by the desirable, continuously predetermined quantitative ratio that it is simultaneous and with time. Although a plating reaction will start again if nonelectrolytic plating liquid is added, the nickel coat formed by adjusting the addition is controllable to desired thickness. After addition termination of nonelectrolytic plating liquid, holding solution temperature for a while, since generating of hydrogen gas is no longer accepted completely, stirring is continued and a reaction is completed.

[0028] Although the above-mentioned nonelectrolytic-plating B process is continuously performed after a nonelectrolytic-plating A process, the approach of classifying a spherical core-material particle and plating liquid, making water newly distributing a spherical core material particle, preparing an aquosity slurry, adding the water solution which dissolved the complexing agent by the density range of 5-50g / L preferably one to 100 g/L there, preparing an aquosity slurry, and performing a nonelectrolytic-plating B process by approaches, such as filtration after nonelectrolytic plating A process termination, is sufficient as it.

[0029] Although formation of a nickel coat and formation of a minute projection are performed by the above-mentioned process on a spherical core material particle, the double layer coat which is further excellent in conductive ability can be formed by performing other metal plating processings (C process) to the front face further. For example, in formation of a golden coat, the nonelectrolytic plating bath which adjusted pH to a complexing agent like EDTA-4Na and citric-acid-2Na and the gold cyanide potassium to the weak acidic field in the sodium-hydroxide water solution is warmed. The mixed

water solution of a gold cyanide potassium, EDTA-4Na, and citric-acid-2Na after adding stirring said nickel-plating powder and considering as distributed suspension. It is performed by actuation of adding and carrying out the plating reaction of the mixed water solution of a boron hydride potassium and a sodium hydroxide according to an individual. Hereafter, it collects as a product by carrying out after treatment with a conventional method similarly. Moreover, the approach of ** and ** can be performed by combining A process and B process like the approach of the above-mentioned **.

[0030] Furthermore, the conductive ingredient which makes conductive nonelectrolytic plating fine particles a conductive filler can be obtained by kneading the conductive nonelectrolytic plating fine particles obtained by doing in this way to the binder which uses insulating resin, such as thermosetting and thermoplasticity, as a principal component, and making them into the shape of the shape of a paste, and a sheet. For example, it is used for the electroconductive glue for carrying out flow adhesion of the connection circuit which counters, the anisotropy electric conduction film, anisotropy electric conduction adhesives, etc. As insulating resin used by this invention, one or more sorts chosen from epoxy system resin, polyester system resin, phenol resin, xylene resin, amino resin, an alkyd resin, polyurethane resin, acrylic resin, polyimide resin, styrene resin, vinyl chloride resin, silicone resin, etc. are mentioned. Moreover, a cross linking agent, a tackifier, a degradation inhibitor, various coupling agents, etc. may be used together if needed.

[0031] The conductive ingredient of this invention can be manufactured by mixing each above-mentioned component. In order to be able to use it with various gestalten, such as the shape of the shape of a paste, and a sheet, and to make it the shape of a paste as a configuration of this conductive ingredient, it can manufacture by containing a solvent suitable in insulating resin. Moreover, in order to make it the shape of a sheet, it can manufacture by applying and drying by bar coater etc. on the polyester system film which performed mold release processing. In the case of-like [paste], this conductive ingredient is used as a connection ingredient which applies on the electrode of the circuit board with a screen printer etc., is made to dry the solvent in insulating resin, forms a 5-100-micrometer paint film, carries out alignment of the electrode of the circuit board which carries out phase confrontation, and makes flow connection with pressurization and heating. In on a sheet, it is used as a connection ingredient which sticks on the electrode of the circuit board, carries out temporary sticking by pressure, carries out alignment of the electrode of the circuit board used as the candidate for connection, and makes flow connection with pressurization heating. the conductor which has an oxide film in the electrode surface which the conductive ingredient obtained above is used for the electrode of a liquid crystal display, connection of LSI for a drive, connection with the circuit board of an LSI chip, etc., and serves as especially a candidate for connection -- it is used suitable for connection between circuits.

[0032]

[Example] Although an example and the example of a comparison are shown and this invention is explained concretely hereafter, this invention is not restricted to the following example.

(Examples 1-5) Making 4.6 micrometers of mean diameters, and the benzoguanamine-melamine-formalin resin [NIPPON SHOKUBAI Make and a trade name "the EPO star"] of true specific gravity 1.4 into a spherical core material, and stirring the 20g to 0.1 g/L palladium-chloride water-solution 400mL, stirring processing was supplied and carried out for 5 minutes, and palladium ion was made to catch. The water solution was filtered, the spherical core material fine particles which carried out repulping rinsing once were supplied, stirring to 1 g/L specific hypophosphite water-solution 400mL of ordinary temperature, reduction processing was performed for 1 minute, and the spherical core material front face was made to support palladium. Subsequently, it supplied in nickel-sulfate water-solution [of the concentration which shows a spherical core material in Table 1 warmed at 60 degrees C], specific hypophosphite water-solution, and 20 g/L sodium-tartrate water-solution 1L, and the nonelectrolytic plating A process was started. It stirred for 20 minutes and checked that foaming of hydrogen stopped.

[0033] after that -- further -- the nickel-sulfate water solution of 224 g/L, and each mixed water solution of the specific hypophosphite of 210 g/L, and the sodium hydroxide of 80 g/L -- judgment addition of the 300mL was carried out through the metering pump at the addition rate for 3mL(s)/, and the nonelectrolytic plating B process was started. Stirring was continued after adding the whole quantity of plating liquid, holding the temperature of 60 degrees C until foaming of hydrogen stopped. Subsequently, plating liquid was filtered, it dried with the 100-degree C vacuum dryer, and the filtration object obtained the fine particles which have a nickel-Lynn alloy-plating coat, after carrying out repulping washing 3 times. Each filtrate after a plating reaction is transparent and colorless, and what the offered plating liquid was completely consumed for by the plating reaction was accepted. It is the spherical particle in which the coat in which all have a minute projection is formed, and it was checked that the plating coat is moreover formed as a dense and substantial continuation coat as it was shown in attached drawing 1 - drawing 3, when the obtained nickel nonelectrolytic plating particle was observed with the electron microscope.

[0034] The electron microscope (SEM) photograph, drawing 2 , and drawing 3 of the resin particle which used drawing 1 for the core material are the SEM photograph of the conductive nonelectrolytic plating fine particles which formed the nickel coat according to examples 1 and 2. It is admitted that a plating layer covers the front face of a spherical core material completely, and the condition of fine particles is moreover presenting the minute projection from these drawings.

[0035]

[Table 1]

例No.	NiSO ₄ (g/1)	NaH ₂ PO ₄ (g/1)
実施例1	2.1	2.3
実施例2	4.5	5.4
実施例3	5.8	15.9
実施例4	7.4	9.0
実施例5	8.9	18.3

[0036] (Example 6) It added stirring to nonelectrolytic plating liquid 750mL of 60 degrees C of solution temperature which adjusted 10g of non-electrolyzed nickel-plating particles obtained in the example 1 to pH6 with the sodium-hydroxide water solution by the presentation which consists of EDTA-4Na (10 g/L), citric-acid-2Na (10 g/L), and a gold cyanide potassium (3.2 g/L, 2.2 g/L as Au), and plating processing was performed for 10 minutes. Subsequently, mixed water solution 120mL of a gold cyanide potassium (20 g/L, 13.7 g/L as Au), EDTA-4Na (10 g/L), and citric-acid-2Na (10 g/L) and mixed water solution 120mL of a boron hydride potassium (30 g/L) and a sodium hydroxide (60 g/L) were separately added in 20 minutes through the liquid-sending pump. Then, liquid was filtered, after carrying out repulping washing of the filtration object 3 times, it dried at the temperature of 100 degrees C with the vacuum dryer, and gilding covering processing (C process) was performed on the nickel-plating coat of a spherical core material particle. A golden coat being dense and being substantially formed as a continuation coat on a nickel-plating coat, was checked without the minute projection formed at the time of nickel plating separating, when the nonelectrolytic plating particle of the obtained double layer was observed with the electron microscope. The electron microscope photograph of the conductive nonelectrolytic plating fine particles obtained at this time was shown in drawing 4.

[0037] (Example of a comparison 1) The powder to which the palladium ion caught on the spherical core material resin particle front face by the same approach as an example 1 was made to return and which back-filtered and gave catalytic activity was obtained. Subsequently, the initial make-up of electrolytic bath of the plating liquid 2L of pH5 which consists of nickel-sulfate 30 g/L, specific hypophosphite 25 g/L, malic-acid sodium 50 g/L, sodium acetate 15 g/L, and lead acetate 0.001 g/L is warmed and carried out to 75 degrees C, the powder which gave the above-mentioned catalytic activity to the bath was thrown in, and stirring distribution was carried out. Adjustment maintenance was carried out at pH which begins pH of a solution by addition of a 200 g/L sodium-hydroxide water solution using automatic regulation equipment during a reaction. Moreover, when the reaction stopped the middle, the specific hypophosphite water solution of 200 g/L was added small quantity every, and the reaction was made to continue. Even if it added the specific hypophosphite water solution, when stopping having foamed, the fine particles which dry all addition at the temperature of 100 degrees C with a vacuum dryer after filtering and carrying out repulping washing of the filtration object 3 times, a stop and, and have a nickel-Lynn alloy-plating coat were obtained. The electron microscope photograph of the obtained nickel nonelectrolytic plating fine particles was shown in drawing 5. Since the product of this example of a comparison was made into the process of the electroless deposition initial-make-up-of-electrolytic-bath method currently held conventionally as I understood from drawing 5, a detailed nickel decomposition product mixes, it is inferior to the adhesion and conductivity of a projection, and practical use could not be presented.

[0038] (Example of a comparison 2) It back-filtered and the powder to which the palladium ion caught on the spherical core material resin particle front face by the same approach as an example 1 was made to return and which gave catalytic activity was obtained. Subsequently, the initial make-up of electrolytic bath of the plating liquid 2L of pH5 which consists of nickel-sulfate 2.1 g/L, specific hypophosphite 25 g/L, malic-acid sodium 50 g/L, sodium acetate 15 g/L, and lead acetate 0.001 g/L is warmed and carried out to 75 degrees C, the powder which gave the above-mentioned catalytic activity to the bath was thrown in, and churning distribution was carried out. Adjustment maintenance of the pH of a solution was carried out by addition of a 200 g/L sodium-hydroxide water solution at the first pH using automatic regulation equipment during the reaction. Moreover, when the reaction stopped on the way, the specific hypophosphite water solution of 200 g/L was added small quantity every, and the reaction was made to continue. Even if it added the specific hypophosphite water solution, when not reacting, the fine particles which dry all addition at the temperature of 100 degrees C with a vacuum dryer after filtering and carrying out repulping washing of the filtration object 3 times, a stop and, and have a nickel-Lynn alloy plating coat were obtained. Since the product of this example 2 of a comparison was the plating particle obtained from the plating bath with low nickel concentration, its plating thickness was thin, and since conductivity was inferior, practical use could not be presented with it.

[0039] (Example of a comparison 3) The powder to which the palladium ion caught on the spherical core material resin particle front face by the same approach as an example 1 was made to return and which back-filtered and gave catalytic activity was obtained. Subsequently, it supplied stirring to 20g / L sodium-tartrate water-solution 2L which warmed the powder which gave the above-mentioned catalytic activity at 65 degrees C, and after fully carrying out stirring distribution and preparing an aquosity slurry, judgment addition of the mixed water solution 320mL of 320mL of nickel-sulfate water solutions of 0.85 mols / L, and the specific hypophosphite of 2.0 mols / L and the sodium hydroxide of 2.0 mols / L was carried out through the metering pump at the addition rate for 5mL(s)/, respectively. Stirring was continued after whole-quantity addition, holding the temperature of 65 degrees C until foaming of hydrogen stopped. Subsequently, plating liquid was filtered, and after carrying out repulping washing of the filtration object 3 times, it dried at the temperature of 100 degrees C with the vacuum dryer, and the fine particles which have a nickel-Lynn alloy-plating coat were obtained. The electron microscope photograph of the obtained nickel nonelectrolytic plating fine particles was shown in drawing 6. Since it manufactured by the approach of electroless deposition continuation dropping excellent in smooth nature that a coat is obtained, the products of the example 3 of a comparison were fine particles without a minute

projection, they are inferior to conductivity and practical use could not be presented with them, so that drawing 6 might show.

[0040] (Physical-properties evaluation) The mean particle diameter of the conductive nonelectrolytic plating fine particles obtained in each aforementioned example and the aforementioned example of a comparison, plating thickness, the adhesion of a projection, magnitude, distribution density, and conductivity were evaluated, respectively, and the result was shown in Table 2. In addition, each physical-properties evaluation was performed by the following approach.

Measurement of the mean particle diameter of plating fine particles: It measured by the Coulter counter method.

Calculation of plating thickness: Nonelectrolytic plating fine particles were immersed in the nitric acid, the plating coat was dissolved, the quantum of the coat component was carried out by ICP or the chemical analysis, and plating thickness was computed by the bottom type.

[0041]

[Equation 1] For the radius (micrometer) of a core material particle, and t, plating thickness (micrometer) and d1 are [$A = [(r+t) 3 - r^3] d_1 / r d^2$] $A = W / 100 - W$, however r / the specific gravity of a core material particle and W of the specific gravity of the plating film and d_2] metal contents (% of the weight).

[0042] Measurement of the adhesion of a projection: Process for 10 minutes with an ultrasonic washing machine (the Honda Electronics Co., Ltd. make, 28kHz, 100W), putting 10g of plating fine particles into a 100mL beaker, adding desalinated water 50 mLs, and stirring with a micro spa tail. After adding desalinated water to the processed slurry and making it 100mL (s), it puts for 10 minutes, supernatant 20mL is taken to a 100mL beaker with a transfer pipet, nitric-acid 20mL is added, and it stirs using a stirring child for 5 minutes. It moved to the 100mL measuring flask, and by ICP, the amount of nickel was measured and the solution which carried out the scalpel rise at 100mL was converted into the amount of nickel per sample 1g (g).

Magnitude of a projection, and measurement of distribution density: The magnitude:plating fine particles which are projections were observed with the electron microscope photograph, the projection looked at by each one plating particle was measured, and the average was taken.

Distribution density: It considered as the average of all the projections that exist on each plating particle in the visual field which can check a projection with an electron microscope photograph.

Conductive measurement: Mix the epoxy resin 100 weight section, the curing agent 150 weight section, and the toluene 70 weight section, and prepare insulating adhesives. Subsequently, the plating fine-particles 15 weight section is blended, and it applies on siliconization polyester film and is made to dry by the bar coating machine. Connection between the glass which vapor-deposited the whole surface with aluminum, and the polyimide film substrate which formed the copper pattern in 100-micrometer pitch was made using the obtained film, and it carried out by the approach of measuring inter-electrode flow resistance. Evaluation is the resistance of 2ohms or less. O It carries out and is 5ohms or more. x It carried out.

[0043]

[Table 2]

No.	平均粒径 (μm)	膜厚 (nm)		尖起物		導電性	
		ニッケル	金	密着性	大きさ (μm)		
実施例1	4.8	94		ND*	0.33	20	O
〃 2	4.8	96		ND	0.40	72	O
〃 3	4.8	93		ND	0.46	92	O
〃 4	4.9	95		ND	0.51	84	O
〃 5	5.1	96		ND	0.63	100	O
〃 6	5.0	81	29	ND	0.38	20	O
比較例1	7.8	72		0.15	0.50	60	X
〃 2	4.7	3		ND	—	—	X
〃 3	4.8	95		ND	—	—	X

* ND : 0.001g/g以下

[0044] It turns out that the conductivity of the example article which satisfies the requirements for this invention as shown in Table 2 is excellent compared with the example of a comparison.

[0045]

[Effect of the Invention] Since the conductive nonelectrolytic plating fine particles concerning this invention have a minute projection in the nickel coat outermost layer and the coat and minute projection are formed as a continuation coat, even if it kneads with matrices, such as synthetic resin and synthetic rubber, a minute projection does not **** or the phenomenon of a coat exfoliating is not produced. In case it is moreover used for electroconductive glue on which the wiring substrate with which the circuit pattern which has an oxide film was formed is pasted up after the circuit pattern has met, especially, good conductive ability can be given and it can apply as a conductive filler as it is. When a gilding coat is furthermore formed on a nickel coat and it considers as a double layer, the engine performance improves further as a conductive ingredient. Moreover, catalyst-ized down stream processing which makes the front face of a spherical core material particle carry out reduction support of the palladium according to the manufacture approach of this invention. After performing catalyst-ized processing, at least the aquosity slurry of an A process:spherical core material Nickel salt, The nonelectrolytic plating process added to the nonelectrolytic plating bath containing a reducing agent, a complexing agent, etc., and B process : The component which constitutes nonelectrolytic plating liquid in the aquosity slurry of a

spherical core material is divided into at least 2 liquid. It becomes possible to produce efficiently above-mentioned conductive nonelectrolytic plating fine particles and an above-mentioned conductive ingredient by performing the nonelectrolytic plating process which adds each that it is simultaneous and with time in a proper combination.

[Translation done.]